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2-[2-(1-BENZOYL-4-METHYLDIBENZO-THIENYL-2)BENZO[*b*]THIENYL-3]PHENYL-1-ETHANONE – A NEW PRODUCT OF THE REACTION OF 1-METHYL-3-PHENYLBENZO-THIENO[2,3-*c*]PYRILIUM PERCHLORATE WITH NUCLEOPHILIC REAGENTS

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On the interaction of 1-methyl-3-phenylbenzothieno[2,3-c]pyrilium perchlorate with primary and secondary amines and alkalis in aqueous media a product of cyclocondensation of 2-acetyl-3-phenacyl-benzo[b]thiophene – 2-[2-(1-benzoyl-4-methyldibenzothienyl-2)benzo[b]thienyl-3]phenyl-1-ethanone – was isolated in addition to the classical objects of recyclization.

Keywords: 2-[2-(1-benzoyl-4-methyldibenzothienyl-2)benzo(*b*)thienyl-3]phenyl-1-ethanone, benzothieno-[2,3-*c*]pyrilium, recyclization, X-ray crystallography.

Recyclization of benzo[*c*]pyrylium salts with primary and secondary amines and alkalis is widely used in preparative chemistry to prepare derivatives of naphthalene [1,2]. Analogous recyclization of benzofuro-[2,3-*c*]pyrylium and benzothieno[2,3-*c*]pyrylium salts with primary and secondary amines allowed the preparation of large series of 4-R-aminodibenzothiophenes and dibenzofurans [3-6]. It is considered that these conversions proceed *via* the ANRORC scheme.

However along with the addition of nucleophiles to pyrylium salts concurrent reactions can occur, linked with 1,1'- and 4,1'-dimerization of pyrylium salts which was first shown by Rostov chemists with the example of the reaction of 1,3-disubstituted benzo[*c*]pyrylium salts with alkalis [7, 8].

The mechanism of 1,1'-dimerization of 1,3-dimethylbenzo[*c*]pyrylium perchlorate is connected with deprotonation of 1-alkyl group with the formation of anhydro base which cannot take part in the formation of classical products of recyclization and tend to dimerize with further conversion into substituted chrysenes. At the same time hydrolysis of pyrylium salts to 1,5-dicarbonyl compounds and their conversion into naphthols also occurs [7].

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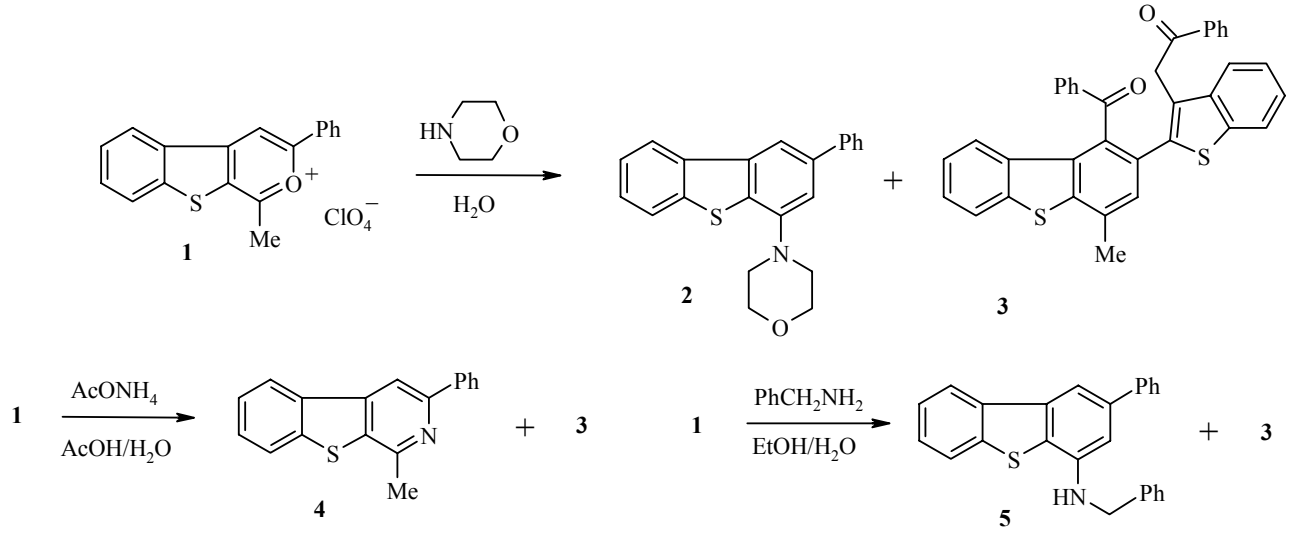
In studying the interaction of 1-methyl-3-phenylbenzothieno[2,3-*c*]pyrylium perchlorate (**1**) with ammonium acetate, primary and secondary amines, we observed that along with the normal products of recyclization which contain in their structure the nucleophile used – 4-morpholino-2-phenyldibenzothiophene (**2**), 1-methyl-3-phenylbenzothieno[2,3-*c*]pyridine (**4**) [9], and 4-benzylamino-2-phenyldibenzothiophene (**5**) [6] – a by-product was formed, the structure of which was not connected with the nucleophile used and which contained no nitrogen according to elemental analysis.



X-ray structural analysis of the compound showed that it was 2-[2-(1-benzoyl-4-methyldibenzothienyl-2)-benzo[*b*]thienyl-3]phenyl-1-ethanone (**3**). In the molecule studied (Fig. 1) the dibenzothiophene unit is not planar, the angle between the benzene rings C(1)···C(6) and C(7)···C(12) is 6°. This is probably explained by intermolecular stacking interactions of base molecule and molecule connected by the symmetry operation (1-*x*, -*y*, 3-*z*) in which only two of the three condensed rings take part (X(1)···X(2)' 3.851, X(2)···X(1)' 3.851, where X(1) and X(2) are centers of the rings C(7)···C(12) and C(1),C(6),S(1)C(7), C(12) respectively, the planes of the

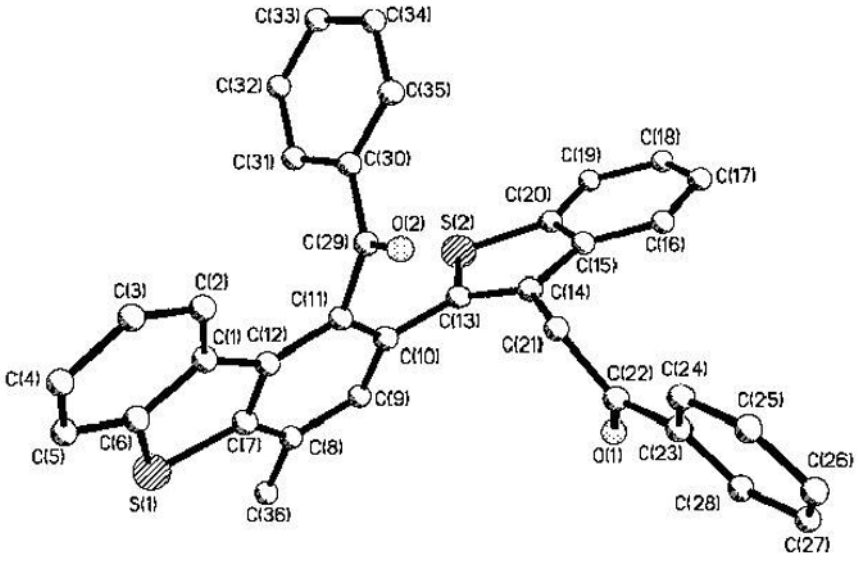


Fig. 1. Structure of the molecule 2-[2-(1-benzoyl-4-methylbenzothienyl-2)-benzo(*b*)thienyl-3]phenyl-1-ethanone (**3**) based on X-ray structural data.

rings are strictly parallel, Fig. 2). The carbon atom of the methyl group C(36) lies practically in the plane of the benzene ring C(7)···C(12) (torsion angle C(36)–C(8)–C(9) 177.8(2)°) while the atom C(29) is lightly deflected from this (torsion angle C(29)–C(11)–C(12)–C(7) 172.2(2)°).The benzothiophene unit is planar with a precision of 0.004 Å.

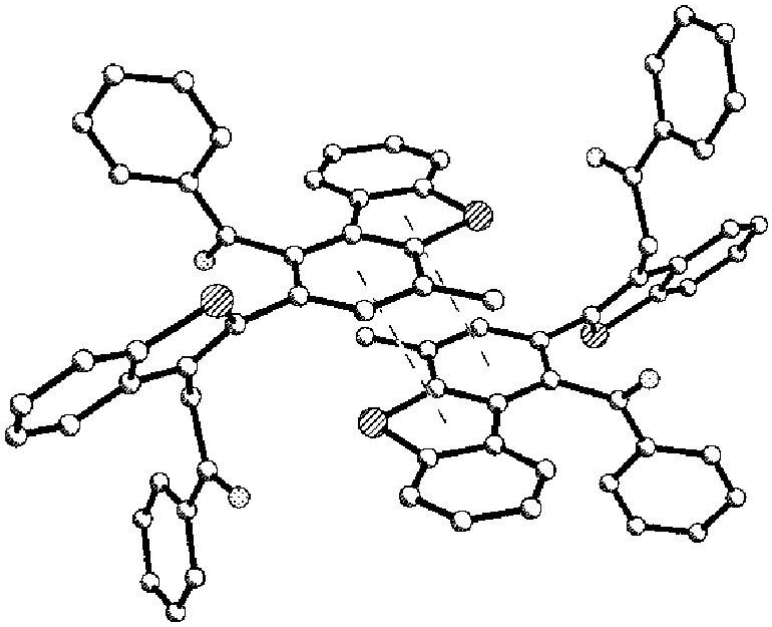


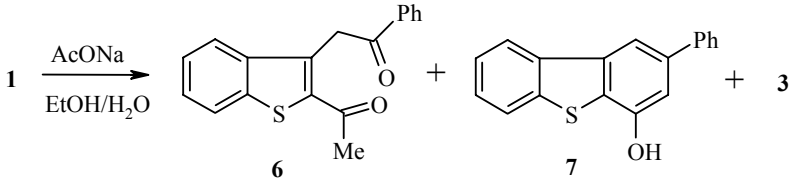
Fig. 2. Dimers in the crystals of compound **3** formed by intermolecular stacking interactions of a molecule with a second molecule connected by the symmetry operation (1-*x*, -*y*, 3-*z*).

The bicyclic and tricyclic fragments are strongly rotated relatively to one another (torsional angle C(9)–C(10)–C(13)–S(2) 59.2(3)°) which results in some lengthening of the C(10)–C(13) bond to 1.476(3) Å (mean bond length 1.455 Å [10]). The oxo groups are somewhat deflected from the planes of the corresponding phenyl substituents (torsion angles O(1)–C(22)–C(23)–C(28) 8.4(4)° and O(2)–C(29)–C(30)–C(35) 18.6(4) Å). The aromatic rings C(30)···C(35) and C(23)···C(28) are practically perpendicular relative to the tricyclic and bicyclic units (angles between the planes are 82.3 and 74.7° respectively) despite the fact that this orientation leads to the shortening of the intermolecular contacts H(24)···H(18) to 2.18 (sum of the van der Waals radii 2.32 [11]) and H(31)···C(11) to 2.59 Å (2.87 Å). In the crystal the molecules of compound **3** form ribbons as a result of very weak hydrogen bonds C(5)–H(5)···O(1)' (1-*x*, -*y*, 3-*z*) (H···O 2.51 Å, C–H···O 153°) and C(17)–H(17)···O(2)' (1-*x*, 1-y, 2-*z*) (H···O 2.53 Å, C–H···O 160°).

We consider that the factor which determines the formation of compound **3** is the presence of traces of water in the reaction medium. In fact recyclization of perchlorate **1** in excess of dry morpholine led to 4-morpholino-2-phenylbenzothiophene (**4**) with minimal impurities of compound **3.** In contrast reaction of perchlorate **1** with morpholine in aqueous alcohol led to preferential formation of compound **3**. We observed analogous behavior on the interaction of perchlorate **1** with a primary amines (benzylamine) in ethanol when the yield of compound **3** varied from 10 to 15%. Similarly recyclization of perchlorate **1** with ammonium acetate in 80% acetic acid gave 30% of compound **3**.

There are no reports in the literature on the formation of compounds of type **3** in reactions of pyrylium salts with nitrogen nucleophiles. In papers on the recyclization of 1,3-dimethylbenzo[*c*]pyrylium perchlorate with alkali the following sequence was described for the formation of chrysenes: pyrylium → dimer → chrysene; and the side process: pyrylium → 1,5-dicarbonyl compound →naphthol. So the authors considered the formation of chrysene from 1,5-dicarbonyl compounds was impossible [7].

Assuming that the mechanism of formation of compound **3** may be analogous to the formation of chrysenes from 1,3-dimethylbenzo[*c*]pyrylium perchlorate, we proposed to obtain dimers of analogous structure from perchlorate **1** under conditions of hydrolysis (sodium acetate–water) as described in [7]. However we isolated a mixture of 2-acetyl-3-phenacylbenzo[*b*]thiophene (**6**) (42% yield), 4-hydroxy-3-phenyldibenzothiophene (**7**) (21% yield), and 2-[2-(1-benzoyl-4-methyldibenzothienyl-2)benzo[*b*]thienyl-3]phenyl-1-ethanone (17% yield).



Further study of the reaction of 2-acetyl-3-phenacylbenzo[*b*]thiophene (**6**) with alkalis, primary and secondary amines showed that just this compound was the precursor of dibenzothiophene **3**. For example, heating compound **6** with aqueous alcoholic KOH gave a mixture of 4-hydroxy-2-phenyldibenzothiophene (**7**) and 2-[2-(1-benzoyl-4-methyldibenzothienyl-2)benzo[*b*]phenyl-1-ethanone (**3**) (44 and 33% yields respectively), and interaction of compound **6** with benzylamine gave a mixture of compound **7** (43), **5** (32), and **3** (11%).

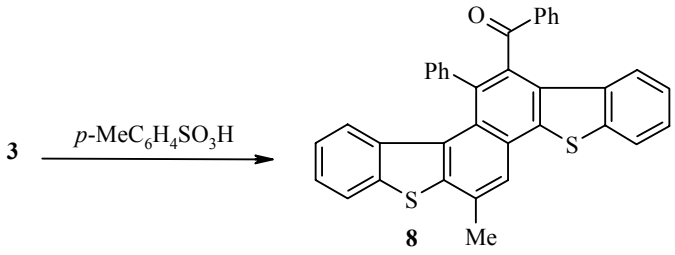


Table 1. Bond Lengths (*l*) in the Molecule of Compound **3**

Bond	*l*, Å	Bond	*l*, Å
S(1)–C(7)	1.741(2)	C(14)–C(15)	1.440(3)
S(1)–C(6)	1.742(3)	C(14)–C(21)	1.491(3)
S(2)–C(20)	1.736(3)	C(15)–C(20)	1.398(4)
S(2)–C(13)	1.744(3)	C(15)–C(16)	1.404(3)
O(1)–C(22)	1.210(3)	C(16)–C(17)	1.372(4)
O(2)–C(29)	1.220(3)	C(17)–C(18)	1.375(4)
C(1)–C(2)	1.397(3)	C(18)–C(19)	1.382(4)
C(1)–C(6)	1.406(3)	C(19)–C(20)	1.392(4)
C(1)–C(12)	1.461(3)	C(21)–C(22)	1.514(4)
C(2)–C(3)	1.361(4)	C(22)–C(23)	1.493(4)
C(3)–C(4)	1.394(4)	C(23)–C(24)	1.387(4)
C(4)–C(5)	1.360(4)	C(23)–C(28)	1.389(4)
C(5)–C(6)	1.398(4)	C(24)–C(25)	1.380(4)
C(7)–C(12)	1.403(3)	C(25)–C(26)	1.368(5)
C(7)–C(8)	1.403(3)	C(26)–C(27)	1.360(5)
C(8)–C(9)	1.374(3)	C(27)–C(28)	1.382(4)
C(8)–C(36)	1.497(3)	C(29)–C(30)	1.481(4)
C(9)–C(10)	1.402(3)	C(30)–C(31)	1.374(4)
C(10)–C(11)	1.394(3)	C(30)–C(35)	1.392(4)
C(10)–C(13)	1.476(3)	C(31)–C(32)	1.391(4)
C(11)–C(12)	1.410(3)	C(32)–C(33)	1.356(6)
C(11)–C(29)	1.508(3)	C(33)–C(34)	1.367(6)
C(13)–C(14)	1.366(3)	C(34)–C(35)	1.360(5)

Table 2. Bond Angles (ω) in the Structure of Compound **3**

Angle	ω, deg	Angle	ω, deg
C(7)–S(1)–C(6)	91.1(1)	C(20)–C(15)–C(16)	118.2(2)
C(20)–S(2)–C(13)	91.7(1)	C(20)–C(15)–C(14)	112.8(2)
C(2)–C(1)–C(6)	117.8(2)	C(16)–C(15)–C(14)	129.0(2)
C(2)–C(1)–C(12)	130.9(2)	C(17)–C(16)–C(15)	119.5(3)
C(6)–C(1)–C(12)	111.3(2)	C(16)–C(17)–C(18)	121.2(3)
C(3)–C(2)–C(1)	120.4(3)	C(17)–C(18)–C(19)	121.3(3)
C(3)–C(2)–C(1)	120.4(3)	C(17)–C(18)–C(19)	121.3(3)
C(2)–C(3)–C(4)	120.8(3)	C(18)–C(19)–C(20)	117.6(3)
C(5)–C(4)–C(3)	121.0(3)	C(19)–C(20)–C(15)	122.2(2)
C(4)–C(5)–C(6)	118.3(3)	C(19)–C(20)–S(2)	126.9(2)
C(5)–C(6)–C(1)	121.7(2)	C(15)–C(20)–S(2)	110.9(2)
C(5)–C(6)–S(1)	125.2(2)	C(14)–C(21)–C(22)	113.1(2)
C(1)–C(6)–S(1)	113.0(2)	O(1)–C(22)–C(23)	120.1(3)
C(12)–C(7)–C(8)	123.0(2)	O(1)–C(22)–C(21)	120.6(2)
C(12)–C(7)–S(1)	113.0(2)	C(23)–C(22)–C(21)	119.3(2)
C(8)–C(7)–S(1)	124.0(2)	C(24)–C(23)–C(28)	117.9(3)
C(9)-C(8)–C(7)	116.6(2)	C(24)–C(23)–C(22)	123.1(2)
C(9)–C(8)–C(36)	121.3(2)	C(28)–C(23)–C(22)	119.0(2)
C(7)–C(8)–C(36)	122.0(2)	C(25)–C(24)–C(23)	121.0(3)
C(8)–C(9)–C(10)	122.6(2)	C(26)–C(25)–C(24)	120.0(3)
C(11)–C(10)–C(9)	120.1(2)	C(27)–C(26)–C(25)	119.9(3)
C(11)–C(10)–C(13)	120.7(2)	C(26)–C(27)–C(28)	120.9(3)
C(9)–C(10)–C(13)	119.2(2)	C(27)–C(28)–C(23)	120.3(3)
C(10)–C(11)–C(12)	119.2(2)	O(2)–C(29)–C(30)	121.8(2)
C(10)–C(11)–C(29)	118.5(2)	O(2)–C(29)–C(11)	120.8(2)
C(12)–C(11)–C(29)	122.2(2)	C(30)–C(29)–C(11)	117.4(2)
C(7)–C(12)–C(11)	118.4(2)	C(31)–C(30)–C(35)	118.4(3)
C(7)–C(12)–C(1)	111.6(2)	C(31)–C(30)–C(29)	121.8(2)
C(11)–C(12)–C(1)	130.0(2)	C(35)–C(30)–C(29)	119.8(3)
C(14)–C(13)–C(10)	129.8(2)	C(30)–C(31)–C(32)	120.3(3)
C(14)–C(13)–S(2)	112.5(2)	C(33)–C(32)–C(31)	120.0(4)
C(10)–C(13)–S(2)	117.7(2)	C(32)–C(33)–C(34)	120.2(4)
C(13)–C(14)–C(15)	112.1(2)	C(35)–C(34)–C(33)	120.5(4)
C(13)–C(14)–C(21)	126.2(2)	C(34)–C(35)–C(30)	120.6(4)
C(15)–C(14)–C(21)	121.6(2)		

Apparently the formation of structure **3** occurs as a result of intermolecular crotonic condensation of two molecules of compound **6**.

Subsequent intramolecular cyclization of compound **3,** in contrast to its benzoid analogs [7], occurs exclusively in acid media, for example, on boiling compound **3** in toluene in the presence of *p*-toluenesulfonic acid.

The results obtained show that the formation of polycondensed compounds on recyclization pyrylium salts with nucleophiles may also occur *via* intermolecular crotonic condensation of two molecules of 1,5-dicarboxylic compounds formed as the result of hydrolysis of the initial pyrylium salts.

EXPERIMENTAL

^{1}H NMR spectra of DMSO-d_6 solutions with TMS as internal standard were recorded on a Gemini-200 (200 MHz) and Bruker DRX500 (500 MHz) spectrometers. The course of reactions and purity of products were monitored by TLC on Silufol UV-254 plates and preparative chromatography on Merck 60 silica gel with chloroform as eluent.

X-ray Crystallographic Study. Triclinic crystals of compound **3**, grown from DMF, $C_{36}H_{24}O_2S_2$, at 20°C: a = 9.843(3), b = 10.968(3), c = 13.349(3) Å, α = 79.88(2)°, β = 79.11(2)°, γ = 81.26(2)°, V = 1383(1) Å^3, M_r = 552.67, Z = 2, space group $P\bar{1}$, d_{calc} = 1.328 g/cm^3, μ(MoKα) = 0.225 mm^{-1}, F(000) = 576. Parameters of the unit cell and intensities of 5191 reflections (4888 independent, R_{int} = 0.035) were measured on a Siemens P3/PC automatic four circle diffractometer (MoKα, graphite monochromator, 2θ/θ scanning, $2\theta_{max}$ = 50°).

The structure was solved by direct methods using the SHELXTL program [12]. Positions of the hydrogen atoms were revealed by an electron density difference synthesis and refined by the "riding" method with $U_{iso} = nU_{eq}$ for the non-hydrogen atom bonded to the hydrogen atom (n = 1.5 for a methyl group and n = 1.2 for the other hydrogen atoms). The structure was refined with respect to F^2 by full matrix least squares analysis in the anisotropic approximation for non-hydrogen atoms to wR_2 = 0.118 for 4829 reflections (R_1 = -0.044 for 2927 reflections with $F > 4\sigma(F)$, S = 1.019). Final atomic coordinates have been deposited in the Cambridge Bank of Structural Data (CCDC 736226). Bond lengths and bond angles are cited in Tables 1 and 2.

Interaction of 1-Methyl-3-phenylbenzothieno[2,3-*c*]pyrylium Perchlorate (1) with Morpholine (General Method). A. A mixture of pyrylium salt **1** (0.8 g, 2.1 mmol) and dry morpholine (4 ml) was boiled for 5 h. The morpholine was removed in vacuum. The residue was triturated with water, filtered, and washed with water to give 4-morpholino-2-phenyldibenzothiophene (**2**) (0.65 g, 90%); mp 122-123°C (ethanol), R_f 0.15 (eluent chloroform). ^{1}H NMR spectrum, δ, ppm (J, Hz): 3.23 (4H, m, CH_2–morpholine); 3.87 (4H, m, CH_2-morpholine); 7.35-7.50 (7H, m, H-7,8, H-C_6H_5); 7.78 (1H, s, H-2); 7.97 (1H, dd, J = 8.4, J = 1.9, H-9); 8.27 (1H, s, H-1); 8.40 (1H, dd, J = 8.4, J = 1.9, H-6). Found, %: C 76.71; H 5.39; N 4.15; S 9.39. $C_{22}H_{19}NOS$. Calculated, %: C 76.49; H 5.54; N 4.05; S 9.28.

B. A solution of pyrylium salt **1** (0.8 g, 2.1 mmol) in ethanol (20 ml) containing morpholine (1 ml) was boiled for 5 h. The solvent was evaporated in vacuum. The residue was triturated with water, filtered, and washed with water. It was purified by column chromatography (silica gel 60, 3×16 cm column, eluent chloroform) to give compounds **2**, **3**, and **7**.

2-[2-(1-Benzoyl-4-methyldibenzothienyl-2)benzo[*b*]thienyl-3]phenyl-1-ethanone (3). Yield 0.35 g (30%); R_f 0.8, mp 198-199°C (DMF). ^{1}H NMR spectrum, δ, ppm: 2.63 (3H, s, CH_3); 4.29-4.41 (2H, m, CH_2); 7.18-7.96 (19H, m, H-arom). Found, %: C 78.49; H 4.47; S 11.42. $C_{36}H_{24}O_2S_2$. Calculated, %: C 78.23; H 4.38; S 11.60.

4-Morpholino-2-phenyldibenzothiophene (2). Yield 0.03 g (4%). Compound **2** was identical spectroscopically and by R_f with the substance obtained by method A and a mixed melting point showed no depression.

4-Hydroxy-2-phenyldibenzothiophene (7). Yield 0.21 g (36%); R_f 0.26, mp 180-181°C. ^{1}H NMR spectrum, δ, ppm: 5.45 (1H, s, OH); 7.1 (1H, s, H-2); 7.33-7.55 (5H, m, Ar); 7.60-7.70 (2H, m, H-5,8); 7.81-7.93 (1H, m, H-5); 7.97 (1H, s, H-4); 8.12-8.24 (1H, s, H-8). Found, %: C 78.45; H 4.52; S 11.39. $C_{18}H_{12}OS$. Calculated, %: C 78.23; H 4.38; S 11.60.

Interaction of 1-Methyl-3-phenylbenzothieno[2,3-*c*]pyrylium Perchlorate (1) with benzylamine and ammonium acetate was carried out by a known method [6, 9].

Interaction of 1-Methyl-3-phenylbenzothieno[2,3-*c*]pyrylium Perchlorate (1) with Sodium Acetate (General Method). A suspension of pyrylium salt **1** (0.8 g, 2.1 mmol) in a solution of AcONa (0.5 g) in 50% 2-propanol was stirred for 24 h at room temperature. The residue was filtered off, washed with water, 50% 2-propanol, and dried. It was purified by column chromatography (silica gel 60, 3×16 cm column, chloroform eluent). Three fractions were collected, the solvent removed at low pressure, to give compounds **3**, **6**, and **7**.

2-[2-(1-Benzoyl-4-methyldibenzothienyl-2)benzo[*b*]thienyl-3]phenyl-1-ethanone (3). Yield 0.1 g (17%); R_f 0.8, mp 198-199°C (DMF).

2-Acetyl-3-phenacylbenzo[*b*]thiophene (6). Yield 0.25 g (42%); R_f 0.52, mp 150-151°C (ethanol). ^{1}H NMR spectrum, δ, ppm: 2.58 (3H, s, CH_3); 5.04 (2H, s, CH_2); 7.19-7.79 (5H, m, H-Ar); 7.81-8.02 (2H, m, H-6,7); 8.02-8.28 (2H, m, H-5,8). Found, %: C 73.59; H 4.61; S 10.57. $C_{18}H_{14}O_2S$. Calculated, %: C 73.44; H 4.79; S 10.89.

1-Hydroxy-3-phenyldibenzothiophene (7). Yield 0.12 g (21%); R_f 0.26, mp 180-181°C.

Compounds **3** and **7** were identical spectroscopically and by R_f with those obtained by interaction of compound **1** with morpholine by method B, and mixed melting points gave no depression.

Interaction of 2-Acetyl-3-phenacylbenzothiophene (6) with Potassium Hydroxide (General Method). A solution of KOH (0.1 g, 1.8 mmol) in water (1 ml) was added to a solution of compound **2** (0.2 g, 0.7 mmol) in methanol (5 ml) and the reaction mixture was boiled for 3 h. It was cooled, diluted with water (50 ml) and acidified with 10% hydrochloric acid until acid. The precipitate was filtered off, washed with water, and dried. The mixture was separated by column chromatography (silica gel 60, column 3×16 cm, eluent chloroform) to give compounds **3**, **7,** and **8**.

2-[2-(1-Benzoyl-4-methyldibenzothienyl-2)benzo[*b*]thienyl-3]phenyl-1-ethanone (3). Yield 0.06 g (33%); mp 198-199°C (DMF).

1-Hydroxy-3-phenyldibenzothiophene (7). Yield 0.08 g (44%); mp 180-181°C. Compounds **3** and **7** were identical spectroscopically and by R_f to compounds obtained by interaction of compound **1** with morpholine by method B and mixed melting points gave no depression.

14-Benzoyl-7-methyl-13-phenylbisbenzo[*b*]thieno[1,2:6,5]naphthalene (8). A solution of compound **3** (0.55 g, 10 mmol) in toluene (10 ml) and *p*-toluenesulfonic acid (10 mg) was boiled for 10 h until complete disappearance (TLC) of the starting compound **3**. The toluene was removed in vacuum. The residue was washed with water and crystallized from a mixture of toluene and hexane. Yield 0.41 g (77%); mp 257-258°C. ^{1}H NMR spectrum, δ, ppm: 2.60 (3H, s, CH_3); 7.18-7.96 (19H, m, H arom). Found, %: C 80.61; H 4.27; S 11.71. $C_{36}H_{22}OS_2$. Calculated, %: C 80.87; H 4.15; S 11.90.

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